LABORATORY DOCUMENTATION REQUIREMENTS FOR DATA VALIDATION

DRAFT

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Quality Assurance Program

USEPA Region 9

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LABORATORY DOCUMENTATION REQUIREMENTS FOR DATA VALIDATION

DRAFT

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INTRODUCTION

In all hazardous site investigations, it is essential to know the quality of the data used for decision-making purposes. The process of generating data of known quality begins in the planning stages when data quality objectives (DQOs) are established (EPA 1993 and 1994); continues during sample collection activities and laboratory analysis; is re-evaluated when validating the analytical data (EPA 1994a, 1994b); and is finalized as part of the data quality assessment process (EPA 1996). This document has been revised to be consistent with the deliverable specifications defined in revisions to contract laboratory program (CLP) inorganic and organic statements of work (EPA 1992, 1994c), and to identify the specific laboratory documentation requirements that are generally necessary as part of the DQO process.

Validation of data requires that appropriate quality assurance and quality control (QA/QC) procedures be followed, and that adequate documentation be included for all data generated both in the laboratory and in the field. Professionals trained in data validation procedures review this information, "flag" data with qualifiers when QA/QC criteria are not met, and prepare the data validation report. The validation reports are then used as sources of data quality indicators, which are used to conduct a data quality assessment relative to the pre-established DQOs.

The QA/QC documentation provided by any laboratory, in conjunction with the sample results, allows for the evaluation of the following indicators of data quality:

- Integrity and stability of the samples;
- Instrument performance during sample analysis;
- Possibility of sample contamination;
- Identification and quantitation of analytes;
- Analytical precision; and
- Analytical accuracy.

The general laboratory documentation requirements discussed in this document are formatted into two (2) sections, pertaining to the organic and inorganic analyses. In addition to the general documentation requirements discussed in this document, several new appendices have been added to provide specific details regarding data deliverables for selected non-CLP analytical methods. The summary of deliverable specifications presented in the appendices were derived for the non-CLP methods commonly used by the EPA Region 9 Analytical Program. The deliverable specifications are not intended to be a prescriptive list that is always required, but rather, they are intended to show what EPA Region 9 has done to establish general analytical documentation requirements in response to the requirements of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

It is hoped that this document will help clarify some of the confusion that exists between the various analytical levels of data quality that were included in the outdated guidance document *Data Quality Objectives fore Remedial Response Activities*. The more recent DQO guidance, *Guidance for the Data Quality Objectives Process* (EPA 1994) requires the use of definitive data for decision making.

Accordingly, any data used to make decisions should be appropriately documented. This document should be used as a menu of quality control parameters, naming conventions, and formatting examples for a specific group of analytical procedures. The fundamental objective is to produce documentation that substantiates data quality.

I. ORGANIC ANALYSES

I.A. Documentation

The data package submitted for EPA data validation will consist of five (5) sections:

- Case narrative;
- Chain-of-Custody documentation;
- Summary of results for environmental samples (including quantitation limits);
- Summary of QA/QC results; and
- Raw data.

I.B. Case Narrative

The case narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or his/her designee. The Case Narrative will consist of the following information:

- Client's sample identification and the corresponding laboratory identification;
- Parameters analyzed for each sample and the methodology used. EPA method numbers should be cited when applicable.
- Whether the holding times were met or exceeded;
- Detailed description of all problems encountered;
- Discussion of possible reasons for any QA/QC sample results outside acceptance limits; and
- Observations regarding any occurrences which may adversely affect sample integrity or data quality.

I.C. Chain-of-Custody Documentation

Legible copies of Chain-of-Custody forms for each sample shall be submitted in the data package. The date of receipt and the observed sample condition at the time of receipt must be described on the Chain-of-Custody form. Copies of any internal laboratory tracking documents should be included.

I.D. **Summary of Environmental Sample Results** (CLP Form I Equivalent)

The following information is to be included in the summary of sample results for each environmental sample. The summary form should follow the CLP format if possible, but other formats are acceptable provided that all necessary information is included.

- Form Title;
- Client's sample identification and the corresponding laboratory identification;

- Sample collection date;
- Sample matrix;
- Date of sample (or sub-sample) extraction and quantity of sample subjected to extraction, as applicable;
- Date and time of analysis;
- Identification of the instrument used for analysis;
- Gas Chromatography (GC) column and detector specifications;
- Weight or volume of sample used for analysis/extraction;
- Dilution or concentration factor for the sample;
- Percentage of moisture in soil sample (optional);
- Method detection limits (MDL) or sample quantitation limits;
- Analytical results and associated units; and
- Definitions for any laboratory data qualifiers used.

I.E. Summary of QA/QC Sample Results

The following QA/QC sample results must be presented on QC summary forms to facilitate data validation and data quality assessment activities. The summaries should follow the CLP format, if possible. Other formats may be acceptable provided that all necessary information is included and the summary is easy to follow. These summaries must have all the information outlined in Section I.D (a detailed summary is presented in Appendix D).

I.E.1. Instrument Calibration (for each instrument used)

Initial Calibration (CLP Form VI equivalent)

Report the analyte concentrations of the initial calibration standards and the date and time of analysis. List the response factor (RF), the average RF, percent relative standard deviation (%RSD), and retention time (for GC analyses) for each analyte. The initial calibration (IC) report must also include a sample identifier (ID), associated injection volume or quantity of sample analyzed, and the acceptance criteria, such as minimum RF values, and associated maximum %RSD values.

Continuing Calibration (CLP Form VI equivalent)

Report the concentration of the calibration standard used for the continuing calibration and for the mid-level standard, and the date and time of analysis. List the RF, percent difference (%D), and retention time (for GC analyses) for each analyte.

I.E.2. Method Blank Analysis (CLP Form IV equivalent)

List the environmental samples and QC analyses associated with each method blank. Report the concentrations of any analytes found in the method blanks.

I.E.3. Surrogate Standard Recovery (CLP Form II equivalent)

Report the name and concentration of each surrogate compound added. List the percent recoveries of all surrogates in the samples, method blanks, matrix spike/matrix spike duplicates and other QC analyses.

I.E.4. **Precision and Accuracy** (CLP Form III equivalent)

- Matrix spike/matrix spike duplicate (MS/MSD) analysis.

Report the name and concentration of each spiking compound. Samples are to be spiked with all specified compounds of potential concern. List the sample results, spiked sample results (concentration and %R), percent recovery and the relative percent difference (RPD). The acceptance criteria must also be presented to facilitate evaluation of both spike and duplicate results.

- Laboratory duplicate analysis, as applicable.

Report the RPD between duplicate analyses, along with the associated acceptance criteria.

- Laboratory QC check sample analysis.

Report the percent recovery for each analyte in the laboratory QC check sample. List the associated acceptance limits.

I.E.5. Other QC Criteria

- GC Retention time windows determination (CLP Form X equivalent)

Report the retention time window for each analyte, for both primary and confirmation analyses.

Retention time windows are established by performing 3 analyses of standards for all analytes being measured throughout the course of a 72-hour period. The retention time window is defined as plus or minus 3 times the standard deviation of the absolute retention time. Retention time windows are to be updated daily.

- Compound identification (GC).

Report the retention times and the concentrations of each analyte detected in the samples for both primary and confirmation analyses.

- MDL determination.

List the method detection limits.

Method detection limits are determined by performing at least 7 analyses of standards for all analytes measured at 2-5 times the required detection limit concentrations. The method detection limits are calculated as 3.143 times the standard deviation of the measured values. Refer to 40 CFR Part 136 Appendix B.

I.F. Raw Data

I.F.1. GC Analyses

This section shall include **legible copies** of the raw data for the following:

- Environmental samples (arranged in sequential order by client sample number);
- Instrument calibrations; and
- QC analyses.

The raw data for both the primary and confirmation analyses are to be included.

The raw data for each analysis shall include the following:

- Chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names);
- Area print-outs or quantitation reports;
- Sample extraction and clean-up logs;
- Instrument analysis logs for each instrument used; and
- GC/MS confirmation, as applicable.

I.F.2. GC/MS Analyses

This section shall include **legible copies** of the raw data for the following:

- Environmental samples (arranged in increasing client's sample number order);
- Mass spectrometer tuning and mass calibration (BFB, DFTPP);
- Initial and continuing instrument calibrations;
- QC analyses;
- Sample extraction and clean-up logs; and
- Instrument analysis logs for each instrument used.

The raw data for each analysis shall include the following:

- Chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names);
- Enhanced spectra of target analytes and tentatively identified compounds (TICs), with the associated best-match spectra; and
- Quantitation reports.

Legible copies of the raw data shall be organized systematically, each page shall be numbered, and a table of contents must be included with each package. The raw data for compound identification and quantitation must be sufficient to verify each result presented in Sections I.D. and I.E.

I.G. SUMMARY OF DOCUMENTATION REQUIREMENTS

Organic Data

Section I. Case Narrative

Section II. Chain-of-Custody Documentation

1. Original Chain-of-Custody forms with ID numbers and laboratory receipt signatures

2. Copies of internal tracking documents, as applicable

Section III. Sample Analysis Results

1. Environmental samples, with quantitation limits

(include dilutions and re-analyses)

Section IV. QC Summary Forms

1. Initial calibration summary
2. Continuing calibration summary
3. Method blank results
4. Surrogate percent recoveries
5. Matrix spike percent recoveries
6. Laboratory duplicate relative percent differences
7. Laboratory QC check sample, if applicable
8. Retention times and acceptance windows
9. Method detection limits (MDLs)

Section V. Raw Data, chromatograms and area/quantitation reports

1. Environmental samples (include dilutions and re-analyses)
2. Instrument tuning, for analyses gas chromatography/mass spectrometry (GC/MS)
3. Initial calibration
4. Continuing calibration
5. Method blanks
6. Surrogate recoveries
7. Matrix spike (MS)
8. Laboratory duplicate or matrix spike duplicate (MSD)
9. Laboratory QC check sample, as applicable

10. Retention time windows

11. Percent moisture for soil samples

12. Sample extraction and clean-up logs

13. Instrument analysis log for each instrument used

II. INORGANIC ANALYSES

II.A. Documentation

The data package submitted for EPA data validation will consist of five (5) sections:

- Case narrative;
- Chain-of-Custody documentation;
- Summary of results for environmental samples (including quantitation limits);
- Summary of QA/QC results; and
- Raw data.

II.B. Case Narrative

The case narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or his/her designee. The Case Narrative will consist of the following information:

- Client's sample identification and the corresponding laboratory identification;
- Parameters analyzed for each sample and the methodology used. When applicable, cite EPA method numbers;
- Whether the holding times were met or exceeded;
- Detailed description of all problems encountered;
- Discussion of possible reasons for any QA/QC sample results outside acceptance limits; and
- Observations regarding any occurrences which may affect sample integrity or data quality.

II.C. Chain-of-Custody Documentation

Legible copies of Chain-of-Custody forms for each sample shall be submitted in the data package. The date of receipt and the observed sample condition at the time of receipt must be described on the Chain-of-Custody form.

II.D. Summary of Environmental Results

The following information is to be included in the summary of results for each environmental sample. The summary should follow the CLP format if possible, but other formats are acceptable provided that all necessary information is included.

- Form Title;
- Client's sample identification and the corresponding laboratory identification;

- Sample collection date;
- Sample matrix;
- Date of sample digestion and quantity of sample subjected to digestion, as applicable;
- Date and time of analysis;
- Identification of the instrument used for analysis;
- Instrument specifications;
- Weight or volume of sample used for analysis/digestion;
- Dilution or concentration factor for the sample;
- Percentage of moisture in the soil sample;
- Instrument detection limits (IDL) or MDL;
- Analytical results; and
- Definitions for any data qualifiers used.

II.E. Summary of QA/QC Results

The following QA/QC sample results must be presented on summary forms to facilitate data validation and data quality assessment activities. These summaries should follow the CLP format, if possible. Other formats are acceptable provided that all necessary information is included and the summary is easy to follow. These summaries must have all information stated in Section II.D.

II.E.1. Instrument Calibration (CLP Form II equivalent)

The order for reporting of calibrations for each analyte must follow the chronological order in which the standards were analyzed.

Initial Calibration Verification

Report the source for the calibrations standards. Report the concentration for the true value, the concentration found, the percent recovery, and the control limits for each element analyzed. The date and time of analysis must also be reported.

Continuing Calibration Verification

Report the source for the calibrations standards. Report the concentration for the true value, the concentration found, the percent recovery, and the control limits for each element analyzed. The date and time analysis must also be reported.

Report results for (low-level) standards used to verify instrument sensitivity (that the reported detection limits can be achieved) in the manner described for continuing calibration verification. This should accompany data packages when modified analytical procedures result in lower reporting (i.e., quantitation) limits.

II.E.2. Method Blank Analysis (CLP Form III equivalent)

Report analyte concentrations found in the initial calibration blank (ICB), the continuing calibration blank (CCB), and in the preparation blank. The date and time of analysis must also be reported.

The order for reporting ICB and CCB results for each analyte must follow the chronological order in which the blanks were analyzed.

II.E.3. ICP Interference Check Sample (CLP Form V equivalent)

Identify the source for the interference check sample. Report the true value, the initial and final results and the calculated percent recovery, and the control limits for each analyte.

II.E.4. Precision and Accuracy

- MS analysis (CLP Form V equivalent)

Report the concentration of the spiked sample result, the sample result and the quantity of spiking solution added to the predigestion spike for each analyte. Calculate and report the %R and list the control limits.

- Post Digest Spike (CLP Form V equivalent)

In addition to matrix spikes, post-digestion spikes are sometimes analyzed during furnace analysis. Report the concentration of the spiked sample result, the sample result, and the spiking solution added for each element when sample matrix conditions require post digestion spikes. Calculate and report the %R and list the control limits.

- Laboratory Duplicate Analysis

Report the original concentration, duplicate concentration and RPD. List the control limits.

- Laboratory Control Sample (CLP Form VII equivalent)

Identify the source for the laboratory control sample. Report the concentration of the spiked sample result, the sample results and the spiking solution added for each element analyzed. Calculate and report the percent recovery and list the control limits.

The laboratory control check sample is prepared following the identical procedure that was used for preparing the analytical samples.

II.E.5. Other QC Criteria

- Method of Standard Additions (MSA)

This summary must be included when MSA analyses are required. Report the absorbance values with corresponding concentration values. Report the final analyte concentration and list the associated correlation coefficient, and the control limits.

- Inductively Coupled Plasma (ICP) Serial Dilution

Report the initial and serial dilution results, the associated %D, and the control limits.

- ICP Linear Ranges

For each instrument and wavelength used, report the date on which the linear ranges were established, the integration time, and the upper limit concentration.

- ICP Inter--element Correction Factors

For each instrument and wavelength used, report the date on which the correction factors were determined. List the inter--element correction factors for Al, Ca, Fe, Mg and any other element and the analytes to which they are applied.

- IDL determination

List the IDLs.

IDLs are determined by multiplying by 3.143, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution at a concentration 3-5 times the estimated detection limit concentrations, with 7 consecutive measurements per day. Refer to the 40 CFR Part 136 Appendix B.

II.F. Raw data

This section shall include **legible copies** of the raw data for the following:

- Environmental sample results (arranged in increasing client's sample number order);
- Instrument calibrations; and
- QC sample analysis data.

The raw data for each analysis shall include the following:

- Measurement print-outs and quantitation reports for each instrument used;
- Absorbance, titrimetric, or other measurements for wet chemical analysis;
- Sample preparation and digestion logs;
- Instrument analysis logs for each instrument used; and
- Percent moisture in the soil samples (when applicable).

Legible copies of the raw data shall be organized systematically, and each page shall be numbered, and a table of contents must be included in each package. The raw data for compound identification and quantitation must be sufficient to verify each result presented in Sections II.D. and II.E.

II.G. SUMMARY OF DOCUMENTATION REQUIREMENTS

Inorganic Data

Section I. Case Narrative

Section II. Chain-of-Custody Documentation

1. Original Chain-of-Custody forms with laboratory ID numbers and sample receipt signatures.

2. Copies of internal tracking documents, as applicable

Section III. Sample Analysis Results

1. Environmental samples, with quantitation limits (include dilutions and re-analyses)

Section IV. QC Sample Result Summary Forms

1. Initial and continuing calibrations

2. Method blanks, continuing calibration blanks, and prep blanks

3. ICP interference check sample

4. Matrix spike

5. Laboratory duplicate

6. Laboratory control sample

7. Method of standard additions

8. ICP serial dilution

9. Instrument detection limits

10. ICP linear range

Section V. Raw Data - sequential measurement readout records for ICP, graphite furnace atomic absorption (AA), flame AA, cold vapor mercury, cyanide, and/or other inorganic analyses.

1. Environmental samples (including dilutions and reanalyses)

2. Initial and continuing calibrations

3. Continuing calibration and Preparation blanks

4. Matrix spikes

5. Post digest spikes

6. Method of standard additions, when applicable

7. Laboratory duplicate or matrix spike duplicates

8. ICP Serial Dilution
9. Laboratory control samples, when applicable
10. Percent moisture for soil samples
11. Sample digestion and/or sample preparation logs
12. Instrument analysis log, for each instrument used
13. Instrument tuning for ICP-MS, when applicable

III. QC REQUIREMENTS SUMMARY

III.A. GC/MS Organic Analyses

QC limits, unless specified below, shall be determined according to the analytical methods and must be consistent with the associated DQOs. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

Presented below is a summary of most analytical requirements for the various QC parameters. In order to document the quality of QC sample results, associated information must also be presented on the QC summary forms (see Appendix B)

1. **Instrument Tuning**

- At the beginning of each day that samples are analyzed.

2. **Initial Calibration**

- At the beginning of the analytical sequence;
- Whenever %D between the continuing and initial calibration response factors exceeds $\pm$25%. This applies to specified compounds of interest, or calibration check compounds (CCCs);
- Whenever the response factors for specified compounds of interest or system performance check compounds (SPCC) are less than 0.300 (0.250 for bromoform) for volatiles or less than 0.050 for semi-volatiles analyses; and
- After installation of a new column or after maintenance service/repair of the GC/MS.

3. **Continuing Calibration**

- Prior to the analysis of environmental samples, on each 12-hour shift that samples are analyzed.

4. **Method Blank**

- Purgeables (Volatiles): After each continuing calibration analysis and after the analyses of unusually concentrated samples, to demonstrate that the system is free of contamination;
- Extractables (Semivolatiles): One for each extraction batch of 20 or fewer samples, for each sample matrix. Analyze method blanks on all instruments used for sample analysis; and

- Method blanks should not contain any analytes of interest and are to be free of interfering peaks.

5. **Calibration Range**

- For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. **Surrogate Standard**

- Surrogate standards (3 for volatiles; 3 phenolic and 3 neutral compounds for semi-volatiles) are to be added to the calibration standards, method blanks, environmental samples and QC samples.

7. **Internal Standard**

- Internal standards (3 for volatiles and 6 for semi-volatiles) are to be added to the calibration standards, method blanks, environmental samples and QC samples; and
- If the extracted ion chromatogram profile (EICP) area for any of the internal standards changes by a factor of two (-50% to +100%) from the last continuing calibration, re-analysis of the samples is required after corrective action.

8. **Matrix Spike (MS) Analysis**

- For each extraction/analysis batch of 20 or fewer samples, for each sample matrix; and
- MS solutions are to contain all specified compounds of interest.

9. **Sample Duplicate or Matrix Spike Duplicate (MSD) Analysis**

- For each extraction/analysis batch of 20 or fewer samples, for each sample matrix.

10. **Laboratory QC Check Sample**

- At the beginning of the QC program and as needed.

11. **Method Detection Limits Determination**

- At the beginning of the QC program and as needed.

III. QC REQUIREMENTS SUMMARY

III.B. Pesticides/PCBs

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. **Initial Calibration**

- At beginning of the QC program;;

- Whenever the %D in calibration factors (CF) between continuing calibration and initial calibration exceeds ±15%; and
- After installation of a new column or after maintenance service/repair of the GC.

2. **Daily Calibration**

- Prior to the analysis of environmental samples, on each day that samples are analyzed.

3. **Mid-level Standard**

- After each group of 10 samples; and
- Report the percent breakdown for 4,4'-DDT and for endrin.

4. **Method Blank**

- For each extraction batch of 20 or fewer samples, for each sample matrix. Analyze method blanks on all instruments used for sample analysis; and
- Method blanks must demonstrate that the analytical system is free of contaminants and interfering peaks.

5. **Calibration Range**

- For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. **Surrogate Standard**

- Surrogate standards are to be added to the calibration standards, method blanks, environmental samples and QC samples.

7. **Matrix Spike (MS) Analysis**

- For each extraction batch of 20 or fewer samples, for each sample matrix; and
- MS solutions are to contain all specified compounds of interest.

8. **Sample Duplicate or Matrix Spike Duplicate (MSD) Analysis**

- For each extraction batch of 20 or fewer samples, for each sample matrix.

9. **Laboratory QC Check Sample**

- At beginning of the QC program and as needed.

10. **Retention Time Windows Determination**

- For each GC column, to be updated daily.

11. **Method Detection Limits Determination**

- At beginning of the QC program and as needed

III. QC REQUIREMENTS SUMMARY

III.C. Purgeable Organics by GC

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. **Initial Calibration**

- At beginning of the QC program;
- Whenever the %D in the CF between continuing calibration and initial calibration exceeds ±15%; and
- After installation of a new column or after maintenance service/repair of the GC.

2. **Daily Calibration**

- Prior to the analysis of environmental samples, on each day that samples are analyzed.

3. **Mid-level Standard**

- After each group of 10 samples.

4. **Method Blank**

- After each daily calibration and mid-level standard analysis and after the analyses of unusually concentrated samples, to demonstrate that the system is free of contamination; and
- Method blanks should not contain any analytes of interest and are to be free of interfering peaks.

5. **Calibration Range**

- For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. **Surrogate Standard**

- Surrogate standards are to be added to the calibration standards, method blanks, environmental samples and QC samples.

7. **Matrix Spike (MS) Analysis**

- For each analysis batch of 20 or fewer samples, for each sample matrix; and
- MS solutions are to contain all specified compounds of interest.

8. **Sample Duplicate or Matrix Spike Duplicate (MSD) Analysis**

- For each analysis batch of 20 or fewer samples, for each sample matrix.

9. **Laboratory QC Check Sample**

- At beginning of the QC program and as needed.

10. **Retention Time Windows Determination**

- For each GC column, to be updated daily.

11. **Method Detection Limits Determination**

- At beginning of the QC program and as needed.

III. QC REQUIREMENTS SUMMARY

III.D. Metals Analyses

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. **Initial Calibration**

- Daily and each time the instrument is set up;
- Whenever the %D between the initial calibration and the continuing calibration exceeds 10% (20% for mercury and graphite furnace atomic absorption [GFAA] analyses);
- Whenever the %D between either of the ICP interference check samples and the true value exceeds 20%; and
- Blank standard required as part of initial calibration.

2. **Continuing Calibration Verification Standard**

- After every ten or fewer samples; and
- Analyses are required to have calibrations with acceptable recoveries (the %D between the initial calibration and the continuing calibration less than 10% [20% for mercury]) before and after the sample analysis.

3. **Blanks**

- Continuing calibration blank run immediately following continuing calibration verification standard; and
- Method blank for each preparation batch of 20 or fewer samples, for each sample matrix.

4. **ICP Interference Check Sample**

- At the beginning and at the end of the analytical run; and

- ICP analyses are required to have both ICP interference check samples with acceptable recoveries (the %D between the true value and the ICP interference check sample less than 20%).

5. **Calibration Range**

- For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. **Matrix Spike (MS) Analysis**

- For each preparation batch of 20 or fewer samples, for each sample matrix; and
- MS solutions are to contain all specified compounds of interest.

7. **Sample Duplicate Analysis**

- For each preparation batch of 20 or fewer samples, for each sample matrix.

8. **Laboratory Control Sample (LCS)**

- For each preparation batch of 20 or fewer samples, for each sample matrix;
- Analyses are required to have the LCS results within acceptable recoveries. The %R should be within the range of 80-120% for all metals.
- LCSs are not usually required for mercury or cyanide determinations, but should these data be deemed to be critical to decision making, it would be reasonable to require the mercury %R to be within the range of 90-110%,cyanide &R to be within the range of 85-115%.

9. **Graphite Furnace Post Digest QC**

- A post digest spike at approximately 10 to 20 µg/L is required for all furnace analyses. If the result is greater than or equal to 10 µg/L in the digestate and the recovery of the spike is not within 85% to 115%, the method of standard additions is required for analyte quantification; and
- If the method of standard additions correlation coefficient is less than 0.995, the method of standard additions analysis is required to be repeated once.

10. **ICP Serial Dilution**

- For each preparation batch of 20 or fewer samples, for each sample matrix, dilute the digestate by five and re-analyze.

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APPENDIX A

DATA DELIVERABLE SPECIFICATIONS FOR NON-CLP METHODS IN THE REGION 9 ANALYTICAL PROGRAM